Partition Behavior of Perfluorooctane Sulfonate (PFOS) and Perfluorooctanoic acid (PFOA) in Riverine Sediments

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Abstract
The sorption of perfluoroalkyl substances (PFASs) on the sediments is an important process which contributes to their fate, distribution and transport in water system. In this study, the sorption kinetic, thermodynamics and effect parameters on partition behavior of perfluoroctane sulfonate (PFOS) and perfluor ooctanoic acid (PFOA) at low initial concentration were investigated. The results show ed that the equilibrium time was achieved within 48h. The sorption isotherms of PFOS and PFOA could be described by Freundlich equation. The surface area and compositi on of sediment particles as well as solution pH values and ion strength strongly influenced the sorption of PFOA and PFOS. Both PFOA and PFOS exhibited higher sorption capacity on the sediments with high organic matter content. These results indicated that multiple driving forces such as hydrophobic exclusion , specific chemical interaction and electrostatic attraction contributed to the nonlinear sorption of PFOA and PFOS on sediments.

Keywords
PFOA; PFOS; sediment; partition properties; natural riverine water

INTRODUCTION
Perfluoroalkyl substances (PFASs) are a new type of persistent organic pollutants (POPs) (Wang & Shih 2011; Wang et al. 2012). They have attracted global concern due to high bioaccumulation, extreme persistence and toxicity, as well as wide distribution in the environment. Of the PFASs, perfluorooctane sulfonic acid (PFOS) and per fluorooctanoic aci d (PFOA), ubiquitously present in the diverse environments (Beskoski et al. 2013; Du et al. 2014), showed the highest persistence and accumulation in the aquatic environment. Thus, PFOS, its salts and perfluorooctane sulfonyl fluoride (PFOS-F) have been added to the Stockholm Convention list of the POPs since 2009 (Wang et al. 2009). PFOA, as a Substance of Very High Concern (SVHC) in candidate list under REACH regulation (ECHA 2015) has been prohibited by the Environmental Pretection Agency of the United State (US-EPA) on a PFOA-stewardship program in 2006(US-EPA 2015).

However, owing to more than 50 years of production and worldwide application, PFOS and PFOA can be released into the natural aquatic environment, causing th e global distribution of these compounds (Zhang et al. 2012). S ubsequently, these pollutants have undoubtedly involved the complex processes such as sorption, degr adation and transport, which determine their environmental fate in aquatic systems (Li et al. 2012). The understanding of these basic mechanisms is of significance in both regulatory and scientific areas. Sediment, with complex compositions including minerals, clay, sand and sedimentary organic matter, is an important sink and reservoir of PFOS and PFOA (Zhao et al. 2014). The sorption of PFOS and PFOA onto the sediment determines their distribution, transport and transformation processes in the aquatic environment. The sorption behavior highly depends on the physical and chemi cal properties of PFOS and PFOA, as well as the conditions of water chemistry such as pH value, ionic strength (IS), and property of dissolved organic matter (DOM). Higgins and Luthy (2006) evaluated various sediment-, solut ion-, and chemical -specific parameters potentially affecting sorption of PFASs.

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They found that organic carbon rather than inorganic oxide content in sediment was the dominant effect parameter on sorption, indicating the importance of hydrophobic interact ions. The similar results were also obtained in other previous studies (Ahrens et al. 2009; Ahrens et al. 2010; Zhao et al. 2012; Zhao et al. 2014; Milinovic et al. 2015), where they reported that hydrophobic interaction predominated the sorption behavior of longer-chain PFASs that were found to adsorb more strongly onto sediment, soil or activated sludge w hile short-chain PFASs were exclusively in water. In addition, previous work had also demonstrated that sorption of PFASs on sediment, soil or sludge increased with increasing solution Ca^{2+} concentration and decreasing pH value, in particular, the sorption behaviors of PFOS was expected to be strongly affected by solution conditions (Higgins 2006; Zhao et al. 2014 ; Milinovic et al. 2015). These observations suggeste d that electrostatic interactions were not negligible in the sorption of PFASs onto sediment. More evidences for both hydrophobic and electrostatic interactions involved in the sorption mechanism of PFASs were found by other researchers using aluminum-rich mineral-humic acid as a simulated sediment and sewage sludges as adsorbents (Zhang et al. 2013 ; Wang et al. 2015). However, to reduce the detection errors most of these studies were performed with high initial concentration s of PFASs ranging from 1 to 1000 μg/L that were much higher than the ir concentrations (ng/L) in the natural aquatic environment (Lein et al. 2008; Dufkov á et al. 2012 Zhang et al. 2012; Anumol et al. 2013).

The sorptive behavior of PFASs onto natural aquatic sediment highly depends on many factors. Particularly with high concentration of PFASs to simulate the natural sediment—water interfacial processes is inadequate and possibly leads to absurd conclusions (Ahrens et al. 2011). For this reason, this study examined the sorption behavior of PFOS and PFOA on riverine sediment using a low initial concentration so as to simulate natural aquatic interfacial process. The specific objectives of this study were : by batch partitioning experiments, to determine the sorption coefficient and isotherms of PFOS and PFOA onto the sediment and to examine the effect of pH, ionic strengths, an d particle size and density of sediment on the sorption. These would provide supplementary data for understanding natural sediment-water interfacial process of pollutants.

MATERIALS AND METHODS
Reagents and chemicals
PFOA and PFOS were purchased fro m AccuStandard Inc (USA). $^{13}C_4$-labelled PFOA and PFOS (Wellington's laboratory) were used as internal standards. Calcium chloride, sodium azide, hydrochloric acid and sodium hydroxide with analytical reagent grade were purchased from local chemical compa nies in Beijing, China. HLPC-grade methanol and ammonium acetate were supplied by Fisher Company (USA). Milli -Q ultrapure water was used throughout the experiment. All standard solutions were prepared in methanol and stored in polypropylene (PP) bottles at 4℃.

Sediment collection and characteristics
Four sediment samples were collected from Daliao River systems, Northeast China. The sampling sites were chosen to reflect a variety of physicochemical properties potentially influencing partitioning of PFOA and PFOS. T he surface sediment s were collected in PP plastic bags using a grab sampler and transferred to the laboratory. The wet samples were air-dried, ground and sieved through 2 mm meshes. Total organic carbon (TOC) in sediments were measured using a h igh temperature TOC analyser (Dohrmann DC -190) after the removal of inorganic carbon by adding diluted HCl until acidification reaction was completed. S pecific surface area (SSA) was analyzed by N_2 sorption method. The pH value of sediment was measured i n a 1:2.5 (w/w) mixture of the sediment with 0.01mol/L of $CaCl_2$ solution by a pH meter. Total Fe and Mn contents in sediment were determined by an ICP -OES Thermo Elemental (TJA) Iris Intrepid spectrometer following digestion of sediment with diluted aqua regia (Li et al. 2012). Sediment particle size fractionation

was conducted by wet sieving the sediments through 40, 80, 120, 240 meshes to obtain three size fractions, i.e. 0.42 ~ 0.20, 0.20 ~ 0.125, 0.125 ~ 0.061 mm. The physicochemical parameters of sediments were shown in Table 1.

Table 1 Physicochemical property of the sediments

Parameters	sediment 1	sediment 2	sediment 3	sediment 4
pH value	7.58	7.38	7.93	7.49
OC (mg/g)	8.09	7.98	7.97	80.4
SSA (m^2/g)	104	66.2	52.1	48.1
Fe (mg/g)	45.8	26.9	19.7	24.3
Mn (mg/g)	0.82	0.38	0.40	0.34

Batch partitioning experiments

Batch experiments included partitioning kinetics, effect of pH value, ion strength and particle si ze fractions, as well as sorption isotherm experiments. The partitioning experiments were conducted in the 50ml polypropylene copolymer (PPCO) Nalgene centrifuge tubes. Duplicate sets of tubes containing 1.0 g (dry weight) of sediment and 30 ml of 0.01mo l/L $CaCl_2$ and 200 mg/L NaN_3 solution were spiked respectively with a certain amount of PFOA or PFOS, and equilibrated on the Thermostatic shaker at 150 rpm and 25°C. To determine an appropriate equilibration time, the centrifuge tubes with initial PFOA or PFOS concentration of 50 ng/L were shaken for 0 ~ 72 hours and periodically removed at selected time intervals of 0, 0.5, 1, 2, 4, 8, 12, 24, 36 , 48, 60 and 72 hours. The effects of pH, ion strength and sediment particle size on partition behavior were investigated by batch sorption equilibration experiment at initial PFOS and PFOA concentrations of 50 ng/L. The pH values of aqueous solution were adjusted in the range of 3 ~ 9 with dilute NaOH or HCl. The concentrations of $CaCl_2$ were 0.001, 0.01 and 0.1mol/L. The sorption isotherm experiments were carried out with PFOS or PFOA concentrations ranging from 5 0 to 500 ng/L and at equilibration time of 48 hours. After shaking, the tubes were centrifuged at 1600r/min for 15min and the supernatants were transferred to the new PP tubes for the extraction and analysis.

For each sample batch, blank samples were prepared using 30 mL of millipore water and treated in exactly the same way as the samples. The control experiment in the absence of sediment showed that the total PFOS or PFOA losses was below 5%, which was neglected in the experiment.

Extraction and determination of PFOA and PFOS

The supernatant was passed thro ugh Oasis HLB cartridge (Waters, 500mg, 6mL) for the extraction of PFOA and PFOS. The cartridges were firstly cleaned by dichloromethane and methanol respectively to remove residual impurities, subsequently preconditioned by 5 mL each of methanol and ultrapure water (Dufkov á et al. 2012; Gong et al. 2016). After extraction, t he cartridges were rinsed with ultrapure water, evacuated to dry for 30 min and eluted with 3 × 5ml methanol into 15ml PP tubes. The elution gathered was dried by Nitrogen blowing, and then 1mL of methanol $-H_2O$ (v:v, 1:3) and 10μL of 150 μg/L internal standard solution were added for UPLC-MS-MS analysis.

The concentrations of PFOS or PFOA were determined by UPLC-Xevo TQD (Waters, USA). An Acquity UPLC® BEH C $_{18}$ column (2.1 ×50 mm, 1.7μm) was used for the separation of analyte. Tandem mass spectrometry was conducted on triple quadrupole mass spectrometer (Xevo TQD, Waters, USA) equipped with an ESI source. The mobile phase s were 2 mmol/L of NH4OAC in water (A) and in methanol (B). The detailed analytical parameters of the target compounds were described in the previous study (Gong et al. 2016).

Sorption data fitting and partition coefficient

Sorption isotherms were constructed by plotting C_{sed} vs. C_w for PFOA or PFOS-sediment batch sorption system. The Freundlich model was used to fit sorption data, as described by equations (1) and (2):

$$C_{sed} = K_f (C_w)^{1/n} \tag{1}$$

or

$$\log C_{sed} = 1/n \log C_w + \log K_f \tag{2}$$

where C_{sed} and C_w are concentrations of PFOA or PFOS in sediment (ng/g) and the solution (ng/L) at equilibrium time; K_f is a Freundlich constant repr esenting the sorption capacity and n is the Freundlich exponent depicting the nonlinearity of sorption.

The interaction of PFOA or PFOS between sediment and water can be described by its partition coefficient (K_d):

$$K_d = C_{sed}/C_w \tag{3}$$

By combination of equations (1) and (3), the K_d values were calculated using the equation (4), which were equilibrium concentration dependent:

$$K_d = K_f(C_w)^{(1-n)/n} \tag{4}$$

Previous studies have shown that the fraction of organic carbon (f_{oc}) has an significant effect on the partition of PFOA or PFOS onto sediment (Higgins and Luthy 2006). Thus, the organic carbon normalised partition coefficient (K_{oc}) was calculated by the following equation (5):

$$K_{oc} = K_d/f_{oc} \tag{5}$$

RESULTS AND DISCUSSION

Sorption kinetics

Figure 1 showed the sorption kinetics of PFOA and PFOS onto sediment. It was obvious that the sorption process could be divided into three apparent stages: a rapid sorption process, a slow sorption process and sorption equilibrium process. The rapid sorption occurred at 0 ~ 10 hours for PFOA and at 0 ~ 5 hours for PFOS. Between10 (or 5) and 40 hours, the sorption rate for PFOA or PFOS gradually reduced, and then equilibrium was achieved after approximately 40 hours for both chemicals.
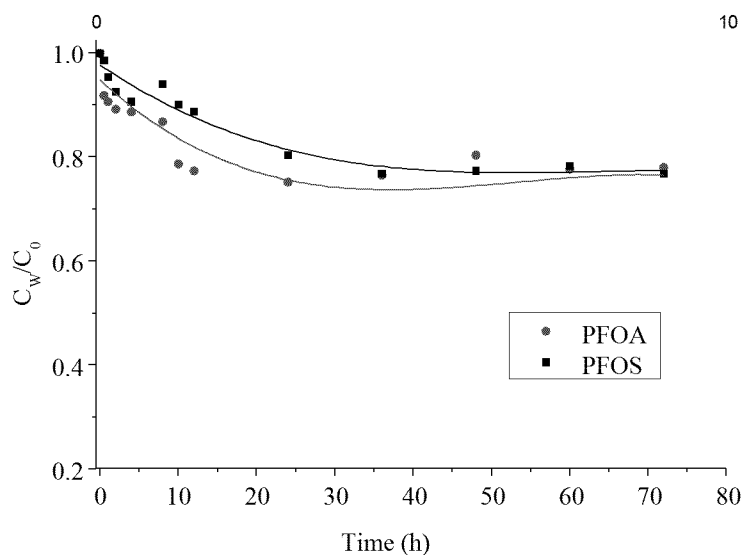

Figure 1. Sorption dynamic curve of PFOA and PFOS onto the sediment（25℃）

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This result was slightly different with the previous study where equilibrium time for PFOS and PFOA was shorter e.g. 4 hours and 8 hours, respectively, when using higher initial concentration (5µg/ L) of PFOS and PFOA (Arvaniti et al. 2014). This indicated that the initial concentration of pollutants was one of impact factors controlling their sorptive kinetics between water and sediment. Simulating natural sedi ment–water interfacial process of pollutants with higher concentration than environmental level would lead to unrealistic results. Therefore, at low concentration of PFOS and PFOA (e.g. 50ng/L) the equilibrium time of 48 hours was adopt ed for the batch sorption system.

Effect of solution pH
The effect of pH value on the sorption of PFOA and PFOS was shown in Fig ure 2 . It was observed that solution pH values in studied range influenced sorption of PFOA and PFOS onto the sediment to som e extent. It was well known that the specific surface species could be formed on the natural sediment surface. Solution pH value would affect specific pH-dependent reactions on sediment surface such as ligand exchange, electrostatic interactions and hydrophobic effect (Zhou et al. 2010; Zhang et al. 2012). Obviously, with increasing pH values between 3 and 7 , the sorption of PFOA and PFOS onto the sediment slightly reduced , which c ould be explained by the electrostatic interaction of PFOA and PFOS anionic species with the positively charged sediment surface at lower pH value. The results were in agreement with Higgins and Luthy's report (Higgins and Luthy 2006). It was also noted that a slight increase in sorption of PFOS and PFOA on the sediment was found at solution pH over 7. Although more negative charges on sediment surface occ urred with pH value increase, resulting in week electrostatic attraction towards PFOA and PFOS anionic specie, ligand exchange might be predominant on interfacial reactions, leading to enhanced sorption of PFOS and PFOA on the sediment (Zhao et al. 2014).
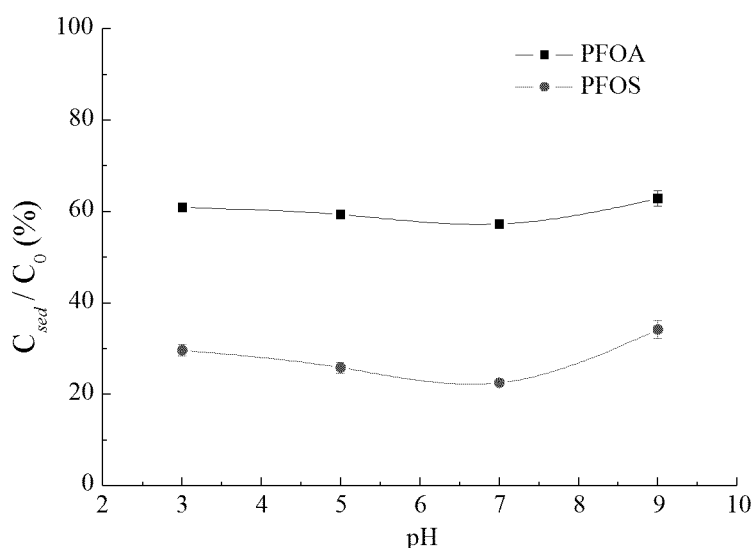

Figure 2. Effect of pH value on the sorption of PFOS and PFOA

Effect of ion strength
Figure 3 revealed the sorption trends of PFOS and PFOA on the sediment with varying in Ca^{2+} concentration from 0.001 to 0.1 mol/L at solution pH value 7. It indicated that the sorption of PFOS and PFOA increased with increasing Ca^{2+} concentration, which was in accord with previous reports by Higgins and Luthy (2006) and by Chen et al (2009). The effect of Ca^{2+} concentration on increased sorption percentage of PFOS and PFOA was likely due to several reasons. One was linked to reduced solubility of PFOS and PFOA as the ionic str ength enhanced, thereby increasing hydrophobic interaction. Chen et al has reported that the sorption -enhancing impact of Ca^{2+}

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concentration was possibly related to the concentration of PFOS and PFOA in the aqueous phase with much stronger impact at low PFOS and PFOA concentration (Chen et al. 2009). Furthermore, with the divalent cation Ca^{2+} increasing, the electrostatic repulsion between anionic PFOS or PFOA molecules and the negatively charged sediment surface was reduced because of neutralization of the negative charge s on the sediment surface by the high Ca $^{2+}$ concentration, which promoted the sorption of PFOS and PFOA on the sediment. Beyond those Ca-bridging effect initiated by cations in the solution needed to be ta ken into consideration (Zhao et al. 2014). It was widely reported that divalent cations have been found to be able to shift the negative sites of adsorbent surfaces into positive ones acting as the bridge to electrostatically attract PFASs (Du et al. 2014). You et al. (2010) also observed that sorption of PFOS on the sediment inc reased when the $CaCl_2$ concentration increased from 0.005 to 0.5 mol/L, suggesting that Ca-bridging effect was responsible for this result. Therefore, the formation of Ca-bridging with sediment might influence the sorption of PFOA and PFOS on the sediment.
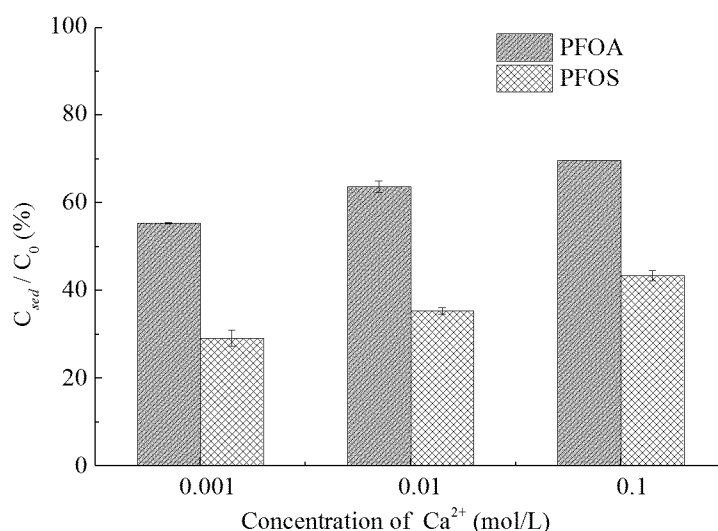

Figure 3. Effect of Ca^{2+} on the sorption of PFOA and PFOS

Effect of particle size

The particle size −dependence of sorption for PFO A and PFOS was shown in Figure 4 . The sorption percentage of target contaminants measured at pH 7 decreased with increasing particle size of sediment. The results might be attributed to specific surface areas and physicochemical properties of different particle size. The specific surface areas were decreased with the increased grain size of sedi ment while the smaller particles would have larger external surface areas (Yu et al. 2009). The di verse compositions and properties of t he fractionated sediment, such as organic carbon fraction , debris of wood and stalks, mineral grains, might result in different sorption capacity to PFASs (Zhao et al. 2012).

On the other hand, the compact organic matter was possibly involved with the smallest sediment particles. It has been repo rted that the organic matter enhanced the sorption capacity of organic pollutants onto sediments (Chen et al. 2009 ; Beskoski et al. 2013 ; Li et al. 2014). As shown in Figure 5, the sorption of PFOS and PFOA on sediments was somewhat related to organic matter, indicating that hydrophobic partitioning played a role in t he interaction between target pollutants and sediment. Therefore, the combination between small -sized sediment and organic carbon was contributed to the sorption of PFOA and PFOS on sediment, which was consisted with the results reported by Zhao et al. (2012).
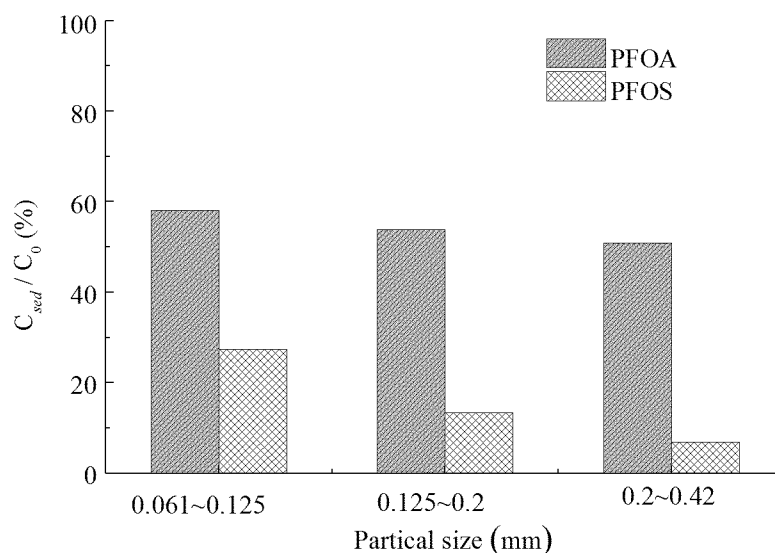
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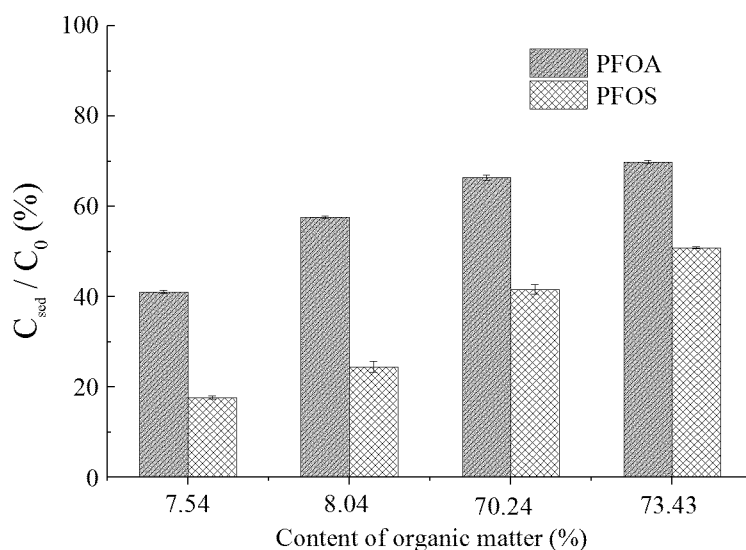Figure 4. Effect of particle size on the sorption of PFOA and PFOS

Figure 5. Effect of organic matter content on sorption of PFOA and PFOS

Sorption isotherms

The sorption isotherms of PFOA and PFOS on sediments were shown in Figure   6.   Equilibrium sorption data was calculated based on the Freundlich model. Over the studied concentrations range of PFOA and PFOS, all experimental data was fitted very well by the nonlinear Fruendlich type sorption isotherm with the regression coefficie nts (R^2) of 0.97 for PFOA and 0.95 for PFOS, respectively.   For both PFOA and PFOS, the sorption coefficients (K_f) were 0.0051 and 0.0012 , and the nonlinearity of sorption   (1/n) were 0.84 and 0.93, respectively.   Regarding nonlinear sorption isotherms,   the partition coefficients K_d and K_{oc} values were   function of the solute concentration, ranged from 0.0012 ~ 0.0027 and 0.15 ~0.36 for PFOA, and 0.00061 ~ 0.00083 and 0.081~ 0.114 for PFOS.

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This nonlinearity of sorption behavior was mainly attributed to organic and mineral matrices involved in sediment . Given that hydrophobicity of perfluorinated chain and hydrophilicity of sulfonate or carboxylate (Zhang et al. 2013), as well as diverse sorption sites on sediments, single driving force seems to be difficult to explain interfacial reaction between pollutants and sediments. The hydrophobic exclusion of perfluorinated chain, specific chemical interaction of sulfonate or carboxylate with sediment, and electrostatic attraction of charged solute molecule with the charged sediment surface might contribute to the nonlinear sorption of PFOA and PFOS , forming the sorption of multi-molecular layers.
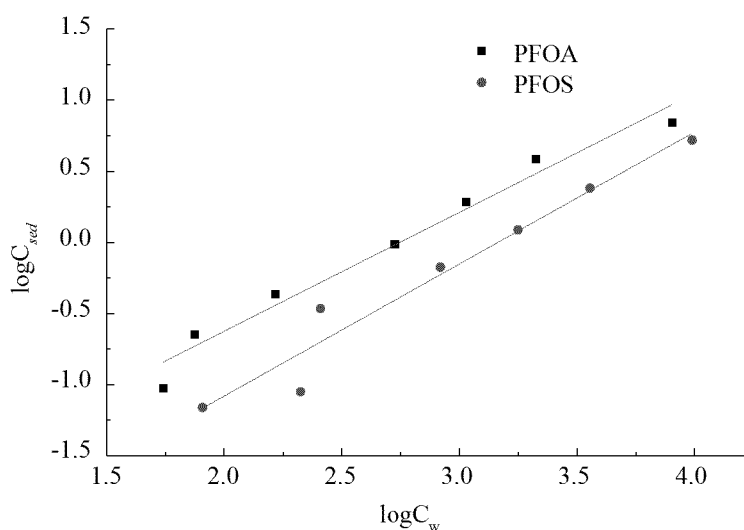

Figure 6. Sorption isotherms of PFOS and PFOA on the sediments

CONCLUSIONS

In natural aquatic system, the sorption of PFOS and PFOA onto the sediments was very low. The kinetic experiments showed that the equilibrium time was achieved within 48h. The sorption behavior of PFOA and PFOS could be described by Freundlich equation. Both PFOA and PFOS displayed higher sorpt ion capacity onto the sediments at high ionic strength and pH values. The sediment particle with small size and high organic matter content enhanced the sorption of PFOA and PFOS due to the surface area and specific composition of particles. These result s had important environmental implications: the leaching of PFCs from sediment would become more serious if background solution was neutral and with low ionic strength. The research would be benefit for the efficient removal of PFOS and PFOA in the natural aquatic systems.

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